QUARTERLY REPORT FOR THE PERIOD
4/1/96 to 6/30/96

Nanostructured Bearing Alloy Studies

[ONR N00014-94-1-0579]
R&T Project Number: MATLSYN 21-01

SUBMITTED TO

OFFICE OF NAVAL RESEARCH
MATERIALS SCIENCE DIVISION
ARLINGTON, VA

ATTN: DR. L.T. KABACOFF

SUBMITTED BY

K. E. GONSALVES
August 10, 1996

INSTITUTE OF MATERIALS SCIENCE
UNIVERSITY OF CONNECTICUT AT STORRS
CT 06269

TEL: 860-486-6134
FAX: 860-486-4745

Quarterly Report April - June 1996

This period marks the transition in the powder making from the Co-Reduction method to the Thermal Decomposition. The latter method was studied earlier in this program and was dropped in favor the Co-Reduction method because of the better chemical homogeneity in powder produced by the Co-Reduction method. The main drawbacks for the Co-Reduction methods for making M50 powder are the high cost of the chemical precursors and the lengthy vacuum sublimation needed to remove the deleterious chloride by-products in the powder. Thermal Decompositon method which does not have these problems, was therefore revisited recently with the goal of improving its homogeneity. It was found that the homogeneity of the Thermally Decomposed powder can be enhanced significantly by high-energy ball mixing method.

Three batches of Thermally Decomposed powder were produced and consolidated during his reporting period. The results, together with those from two batches of Co-Reduced powder processed recently, are given in the following table.

Batch No.	Sublimation Conditions	Hydrogen Treatment	Consolidation Conditions	Oxygen Wt. %	Carbon Wt. %	Hardness RC
			Co-Reduced Powder			
44	700C/2h/vac.	700C/2h/H_2	1000C/413 MPa 40 Min.	3.00	0.73	38
45	700C/2h/vac.	700C/2h/H_2	1000C/413 MPa 40 Min.	3.30	4.50	41
			Thermally Decomposed Powder			
43	Not Needed	400C/2h/H_2	1000C/413 MPa 40 Min.	2.90	0.01	38
46	"	400C/1h/H_2	1000C/413 MPa 40 Min.	0.86	1.20	38
47	"	400C/1h/H_2	750C/413 MPa 120 Min.	3.80	2.90	67

Several interesting points can be observed from the results presented:

Hardness: Hardness of the M50 powder compacts appears to be governed by the consolidation temperature, independent of the chemical methods used for synthesizing the powder. Higher hardness is achieved by consolidation at a lower temperature. Further, by comparing the hardness and microstructure of Batch 47 compact with those consolidated previously at the same temperature, it appears that the application of high-energy ball mixing to Batch 47 powder had increased the hardness of the compact and made the consolidation of the powder more difficult as evidenced by the presence of the micron sized pores in this compact. Such pores are absent in previous compacts consolidated at the same temperature from powders without the ball mixing.

Variation in Carbon and Oxygen Contents: Results from the Co-Reduced Batch 44 and 45 powder compacts show that powders with apparently similar heat treatment can results in compacts with significant difference in carbon content. Similar variation can also be observed in the Thermally Reduced Batch 46 and 47 compacts. To determine the cause for the variation, the carbon content in the powder will be determined at various stages during the processing cycle including in the as-received condition, after hydrogen treatment and after consolidation.

Nanostructured Bearing Research Group
Quarterly Report Apr - Jun 1996

John E. Morral and Stephan F. Schady

Experimental Procedure

Compacts 43 - 46 were prepared during the current quarter. All samples were studied during this quarter, as long as they could be sectioned and were uncontaminated. These specimens were sectioned into individual samples for TEM, SEM, chemical analysis, Vickers microhardness, XRD, dilatometry, and compression testing.

A post-consolidation heat treatment was performed in which samples were quenched from above the critical austenitizing temperature.

Results and Discussion

TEM studies indicated that all samples produced in this quarter were microcrystalline because of the 1000 C sintering treatment. Therefore the results can be used to compare microcrystalline M50 prepared by chemical processing with conventional M50.

The stress strain curve for conventional M50 showed a finite yield strength and work hardening regime. However, the samples produced during this quarter showed a zero initial yield strength that increased with deformation. Also, the measured elastic modulus was about 20 % lower than conventional M50. Heat treatments of the samples after compaction both hardened and strengthened the samples in a way expected for conventional M50.

X-ray diffraction proved unreliable in the determination of grain size. Peak broadening was observed even though the samples were microcrystalline. Additional broadening after the post-consolidation heat treatment presumably was a result of transformation to martensite. Optical microscopy demonstrated that samples sintered at 1000 C show a precipitation structure on a 1-10 micron scale which is further evidence that the grain size is not nanocrystalline.

Conclusions

The higher temperature consolidation (1000 C) was done in order to lower oxygen content, but causes grain growth into the micron range. There is strong evidence that all samples produced during this quarter are microcrystalline and these samples showed a marked difference in mechanical behavior to microcrystalline conventional M50. Presumably this difference is due to porosity and chemistry differences from conventional M50.

Future Work

Determining the source of the oxygen and carbon content in the samples and the effects of the argon gas used in the press will be studied.

Heat treatments for optimum processing of the powders and compacts will be investigated including the determination of the minimum austenitizing temperature to prevent against grain growth and the inclusion of a tempering step in the treatment to increase toughness and strength.

The lower temperature compacts will be investigated and their properties will be compared to commercial grade M50 and to the microcrystalline M50 that was produced in this program in order to determine properties unique to the nanocrystalline material.